

DEVELOPMENT, FABRICATION, AND DELIVERY  
OF NEODYMIUM DOPED YAG  
LASER RODS

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Huntsville, Alabama 35812

By: Howard M. Dess  
Crystal Products Research and Development Department

UNION CARBIDE CORPORATION  
Linde Division  
Speedway Laboratories  
P. O. Box 24184  
1500 Polco Street  
Indianapolis, Indiana 46224

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## I. ABSTRACT

The melt pulling technique developed prior to the start of this program has been scaled up and improved to permit the routine growth of high quality Nd:YAG crystals in sizes ranging up to 0.65-0.75 inch in diameter by 3-3.5 inches long. Even in the largest of these specimens, the central core region can, under appropriate control conditions, be maintained straight and narrow enough to permit fabrication of core-free 1/4-inch diameter laser rods.

Extended annealing tests at 1850-1870°C (approximately 100°C below the melting point of YAG) have not succeeded in eliminating the core. Moreover, similar tests on core-free Nd:YAG rods did not result in any significant improvement in quality as measured by passive optical tests.

A total of 20 Nd:YAG laser rods (with dielectric end coatings) were shipped to MSFC. These rods ranged from 3 mm to 6.38 mm in diameter and 30 mm to 75 mm long. The rods cut from the largest diameter boules generally showed the best passive optical properties. This is attributed primarily to the more favorable growth conditions existing in the larger stations.

The laser thresholds measured were greatly affected by the type of end configuration applied to the rod. Thus, for equivalent size rods, flat and parallel ends normally yielded much higher thresholds than 12" radius ends, and the difference was significantly greater in the 75-mm rods than in the 50-mm long rods. On the other hand, no obvious correlation could be found between the threshold values and the passive optical measurements recorded for the same rods.



## II. INTRODUCTION

The exceptionally favorable characteristics of the trivalent neodymium ion for four-level laser action were recognized at a relatively early stage of the search for solid state laser systems that was triggered by the discovery of the ruby laser. Thus,  $\text{Nd}^{+3}$  was known to exhibit a satisfactorily long fluorescence lifetime, narrow fluorescence linewidths, and possessed a terminal state for the laser transition sufficiently high above the ground state so that CW operation at room temperature was readily feasible.<sup>(1)</sup> Therefore, this ion was incorporated as a dopant in a variety of host materials, e.g., glasses<sup>(2)</sup>,  $\text{CaWO}_4$ <sup>(3)</sup>,  $\text{CaMoO}_4$ <sup>(4)</sup>,  $\text{CaF}_2$ <sup>(5, 6)</sup>,  $\text{LaF}_3$ <sup>(7)</sup>, etc., in an effort to make practical use of its great laser potentialities. However, most of these early hosts displayed undesirable shortcomings either from the standpoint of their intrinsic physicochemical properties or because of the way in which they interacted with the  $\text{Nd}^{+3}$  ions. Glass, for example, broadens the fluorescence linewidth. The tungstates and molybdates pose growth problems because of the necessity for charge compensation when trivalent neodymium substitutes for divalent calcium; moreover, crystals of these compounds do not possess sufficient mechanical strength to withstand easily the rigors of fabrication or long-term operation as lasers. The fluorides have similar problems plus the additional disadvantage of reactivity with moisture or oxygen. Finally, yttrium aluminum garnet ("YAG") was explored as a host for  $\text{Nd}^{+3}$  and its superiority to others which had been tried was quickly demonstrated<sup>(8)</sup>: thus, Nd:YAG lasers displayed the lowest thresholds for CW operation at room temperature of any known host/dopant combination. Furthermore, YAG is physically strong, chemically stable, and possesses desirable thermal properties (e.g., high melting point, high thermal conductivity, low coefficient of thermal expansion) so that, overall, Nd:YAG laser rods are rugged and durable under a wide range of use conditions.

The initial laser tests on Nd:YAG were carried out with crystals that had been grown by three different techniques: the Verneuil, flux, and Czochralski methods (the latter were supplied by the Linde Crystal Products Department)<sup>(8)</sup>.

The quality of the flux-grown and Czochralski crystals was about equivalent but both were superior to the Verneuil material. However, although flux growth is capable of yielding very high quality crystals, this technique affords little control over the shape and size of the crystals, and moreover is very time consuming. The Czochralski process is not subject to these limitations and is broadly applicable to the growth of any material such as YAG which fulfills the following minimum requirements: (1) melts congruently, (2) undergoes no disruptive phase transitions below the melting temperature, (3) the vapor pressure of all components in the melt is acceptably low, and (4) the melt can be contained stably in a refractory metal crucible. These favorable technical factors, plus the availability in the Crystal Products Department of an existing reservoir of practical Czochralski know-how originating from the on-going ruby program facilitated rapid progress in the development of a Czochralski-YAG capability.

The state-of-the-art of YAG growth at the beginning of this contract was as follows: good quality 3/8-inch by 2-inch long crystals of Nd:YAG were being grown routinely. These were sufficiently large to permit fabrication of 3-mm diameter by 50-mm long rods. The length was limited by the amount of melt which could be conveniently accommodated in the growth system then in use. However, the rod diameter was subject to a more fundamental limitation, and this was the occurrence of a central core region of gross optical inhomogeneity in all as-produced crystals. This core is now known to result from a local enrichment in neodymium concentration which in turn is caused by the presence of characteristic facets on the growth interface. High quality Nd:YAG rods could thus only be fabricated from the core-free region of a crystal. However, the quality level was still below that of the best ruby, as measured by passive optical techniques such as Twyman-Green interferometer examination, schlieren photographs, and beam divergence (or low angle scattering) measurements.

The present program grew out of a need to obtain Nd:YAG laser rods especially suited for CW operation in communication experiments, e.g., rods displaying low threshold, high gain, and low laser beam divergence. For high gain, a rod length

of at least 75 mm was considered desirable although a maximum diameter of about 6 mm was specified to assure uniform pumping throughout the rod. Furthermore, to achieve the low threshold and beam divergence, crystals with high optical quality are needed. These requirements were translated into the following specific program objectives:

1. Scale up the growth stations to permit growth of Nd:YAG crystals 3-4 inches long by at least 5/8 inch in diameter.
2. Improve the optical quality of the Nd:YAG crystals by better control of growth parameters.
3. Determine whether annealing treatments might further improve optical quality of as-produced Nd:YAG crystals or fabricated rods.
4. Monitor the effects of the above by extensive evaluation tests, both passive and active.

Finally, during the course of the contract, a total of twenty Nd:YAG laser rods of various sizes in the range 3- to 6-mm diameter by 30- to 100-mm long, with suitable end finish and dielectric coatings was to be delivered to MSFC for laser experiments.

### III. EXPERIMENTAL RESULTS

#### A. Growth Studies

During the course of this program, major effort was focused on learning how best to increase the size of the Nd:YAG crystals produced while at the same time improving the quality. Previous experience with the growth of Nd:YAG crystals strongly pointed to the immediate need for increasing the melt volume for two reasons primarily:

1. The segregation coefficient for  $\text{Nd}^{+3}$  in YAG is less than one, and therefore this dopant ion tends to concentrate in the melt. If too large a fraction of the melt is removed as a result of crystal growth, undesirably large  $\text{Nd}^{+3}$  gradients would be expected to form.
2. The quality of Nd:YAG crystals produced is highly sensitive to the thermal stability of the system. Evidently relatively minor temperature fluctuations can result in the formation of local defects such as neodymium rich precipitates, or bubbles which not only act as scattering sites but also cause severe internal stresses. A large melt-to-crystal volume ratio is preferred because it tends to enhance the thermal stability of the system.

Therefore, a new growth station was built which could accommodate larger crucibles than were currently in use. Although the design of the new station was largely patterned after that of the older ones, nevertheless, the operating characteristics had changed sufficiently so that in effect a series of calibrating runs had to be carried out in order to optimize control of the growth process. This necessitated studying the effect of systematic changes in such growth parameters as rotation rate, pull rate, thermal gradients in the growth chambers, etc.

The first successful size increase was accomplished early in the program when Nd:YAG crystals 5/16 inch in diameter by about 3-1/2 inches long were produced; these are large enough to permit fabrication of core-free 3 mm x 75 mm laser rods. The upper crystal in Figure 1A shows the typical appearance of such a crystal. The problems associated with the growth of this size crystal are primarily those arising from the very slow, steady growth rates that were found to be absolutely essential for the production of highest quality material. This posed a severe test of both equipment reliability and operator judgment during the growth runs which were of necessity continued on an around-the-clock basis for the better part of a week. However, sufficient control and experience were gained over the system so that very soon these longer crystals were being produced on a routine basis.

The next stage in scale-up is represented by the lower crystal shown in Figure 1A. This crystal is in a size range large enough to permit fabrication of 5-mm diameter by 50-mm long laser rods that are free of distortion resulting from the central core region. The final stage of scale-up investigated during the course of this program yielded crystals in the size range shown in Figure 1B, about 0.5-0.75 inch in diameter by 3 to 3-1/2 inches long and capable of yielding 1/4-inch diameter core-free rods 1-1/2 to 2 inches long, as well as any of the smaller diameter rods desired. This largest size crystal was deemed well worthy of the development effort required because the initial products obtained indicated that the core-region could be maintained nearly constant in size despite the overall increase in crystal diameter (see Figures 2 and 3). The disturbing effects of the core on the outlying material were thus more effectively localized, and it was possible to obtain rods of much higher optical quality from such crystals. However, the difficulties encountered in producing these largest Nd:YAG crystals were greater than anything experienced in the growth of the smaller specimens and work is still in progress aimed at learning how best to optimize the growth process. Some of the more troublesome problems associated with the growth of the largest Nd:YAG crystals were the following:

1. Inadequate control by operators. During the initial growth trials of the large crystals, the operators had problems gauging the response of the crystal to changes in furnace conditions. This resulted in marked diameter fluctuations which in turn were associated with the appearance of local scattering sites and strained areas. As the operators became more familiar with the behavior of the system, they learned to take anticipatory action that past experience showed was necessary at various specific stages of growth. The wide diameter fluctuations were thus effectively eliminated.

2. Cracking

YAG's favorable combination of mechanical and thermal properties make it relatively resistant to thermal shock damage both during growth and in most lasing situations. However, during an early phase of the work on the largest size crystals, cracking occurred with disturbing regularity, normally within about one minute after shut down, although one crystal cracked during growth shortly before it was scheduled to be pulled from a melt. Utilization of very slow initial cool-down periods, after the crystals were pulled from the melt did not stop the cracking. Closer examination of the crystals then revealed that the cracks always passed through, and probably originated at, a type of surface irregularity that was characteristic of this series of runs. These defects were roughly hemispherical depressions, 1 to 3 mm in diameter, numbering perhaps two or three, and found only after 2 to 2.5 inches of growth. Further study showed that these defects were caused by attachment to the crystal growth interface of small bubbles that somehow formed in the melt. There they

interfered with growth, resulting in the kind of depressions described above. Although the precise origin of these bubbles is still a matter of speculation, their formation was suppressed by suitable modifications in charging and melting techniques. Subsequently, cracking of the larger crystals has been eliminated as a problem.

### 3. System Reliability

The growth of these largest size Nd:YAG crystals requires still longer times and more exacting control conditions than have been necessary for any of the smaller sizes. By the same token, as run times stretch out, the greater becomes the probability that some random upset will be introduced into the system from the outside, or that some critical operating or control component will fail causing premature termination of the run. Therefore, considerable effort has been expended in adding safeguards to the growth station that would help shield the system against externally originating fluctuations; and all weak or failure-prone components have been replaced or up-graded as rapidly as possible. However, this latter stage of the program still has not been carried to what is considered the ultimate stage of optimization, and additional work will be required beyond the scope of the present program to achieve this objective.

## B. Annealing Studies

Annealing studies on Nd:YAG were of interest for two primary reasons: first, it was vitally important to learn whether the central core area in the as-produced crystals could be eliminated by annealing. Second, it was hoped that annealing of fabricated core-free rods might yield lower residual stress levels in the final products.

Initial tests were conducted on whole crystal specimens in an evacuated tantalum-resistance furnace. No obvious diminution in strain levels was observed (visual inspection under crossed polars) after the crystals had been fired to an estimated 1860° for 2 hours followed by a gradual shutdown. However, temperature measurement and control were known to have been unsatisfactory and, moreover, the heating in vacuum caused complications due to color formation in the crystals. Therefore, it was considered desirable to carry out additional tests in a more quantitative manner on fabricated rods whose optical properties could be well characterized by standardized procedures. A set of YAG rods, both Nd-doped and undoped, some containing tubes and others tube-free, were subjected to the same annealing cycle that earlier proved satisfactory for removal of residual stress in calcium molybdate (samples heated up at 200°C/hr to 1380°C under O<sub>2</sub> atmosphere, held at 1380°C for 10 hours, cooled down at 100°C/hr to 1200°C, then furnace shut off). As was reported in detail in the Second Quarterly Report, passive optical evaluation tests carried out before and after annealing revealed no detectable improvement in quality in any of the rods. In view of the great stability of the YAG lattice, this result is not too surprising. In such materials, significant amounts of atomic migration, necessary for stress relief, cannot be expected to occur in relatively short periods of time (i. e. , hours) except at temperatures not far removed from the melting point. Experience with ruby suggested that the annealing temperature should be within about 100°C of the melting point. Therefore, two of the core-containing rods that had previously been annealed at 1380°C (one was undoped and the other



contained 1.3 at % Nd) were reheated under a nitrogen atmosphere\* to 1850°C over a period of 5 hours, then held in a range 1850-1870°C (uncorrected pyrometer readings) for a total time of 40 hours. At the end of this time, the furnace was cooled 100°C/hr to 1400°C, then shut off. The photographic record (see Third Quarterly Report) of Twyman-Green interferometer measurements and passive far field patterns taken before and after annealing showed no detectable changes. Thus, it was not feasible to eliminate the core region from as-grown crystals by annealing.

The effect of annealing on core-free fabricated rods that were already of high quality was next investigated. The same heat-up and cool-down schedules were applied via the induction furnace set-up described above; however, the rod was held at 1850°C for a period of only 5 hours. As was shown in the Third Quarterly Report, although the before-and-after-annealing Twyman-Green photographs and far field photographs appeared identical, the passive beam divergence appeared to have improved significantly. Therefore, an attempt was made to reanneal the same rod at 1900°C. However, a temporary overshoot in temperature resulted in meltdown. Fortunately a twin to the first rod that was lost was available, both having been fabricated from opposite halves of the same crystal. This second rod, 1493A, was held for a total of 41 hours in the temperature range 1850-1870°C. As is shown by the data in Figure 4, there was no perceptible improvement in rod quality as a result of this longer term annealing treatment. Especially to be noted is the fact that the before-and-after passive beam divergence measurements for this rod are essentially within the experimental error of the procedure used. The apparent improvement in passive beam divergence recorded for the first rod is thus open to question. A possible error in measurement is suspected.

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\* Because a different furnace setup was used. The higher temperature range was achieved by inductively heating a thermally insulated iridium sleeve within which the specimens were mounted. A nitrogen atmosphere was maintained in the furnace to suppress oxidation of the iridium.

Close inspection of Rod 1493A under an intense beam of well collimated light revealed the presence of a number of scattering sites. Because of the small size of these centers, microscopic examination was incapable of yielding further information as to their nature (i. e., were they bubbles or solid inclusions?). End-on observation of the rod through crossed polars clearly showed a striated strain pattern thought to be associated with the disturbing influence of these scattering sites on the local lattice environment. Thus, unless the annealing treatment could result in the elimination of these scattering sites, no significant change in optical properties due to stress relief would be expected. A similar argument is equally valid for the case of the cored rods, except here the much larger optical disturbances result primarily from the presence of the sharply increasing neodymium concentration gradients near the central growth axis of the crystals. Annealing would not be expected to improve the optical quality of cored rods unless significant amounts of atomic migration occurred which would serve to level these Nd-gradients.

### C. Description and Evaluation of Rods Produced

During the course of this program, a large number of growth experiments were carried out under a rather broad range of conditions in an effort to fulfill the objectives agreed upon and outlined in the contract. A certain fraction of these runs had to be terminated at an early stage because of some obvious system malfunction. In other cases, runs were carried through to completion but visual inspection of the crystals would easily reveal grossly defective regions (e. g. large clumps of bubbles, pronounced strain flares emanating widely from the core region, etc.) so that fabrication of rods was obviously not worthwhile. The remaining crystals were generally of high enough quality to justify fabrication of rods for fulfillment of the 20 that were to be supplied to MSFC and for the annealing tests. Table I contains a complete listing of the various rods that were shipped. The 3 mm x 30 mm rods were shipped early in the program, during the first quarter in fact, since it was felt that the quality of these rods was already fairly typical of what could be produced in this size range.

TABLE I

SUMMARY OF Nd:YAG<sup>(1)</sup> RODS SHIPPED TO MSFC  
UNDER THE PRESENT CONTRACT<sup>(2)</sup>

<u>Dimensions</u>		<u>Type of End</u>	<u>Number</u>
<u>Diam. (mm)</u>	<u>Length (mm)</u>	<u>Finish</u>	
3	30	Spherical	2
3	30	Flat and Parallel	2
3	50	12" Radius	2
3	50	Flat and Parallel	2
3	75	12" Radius	5
3	75	Flat and Parallel	2
5	50	12" Radius	2
5	50	Flat and Parallel	1
6.35	38 <sup>(3)</sup>	12" Radius	1
6.35	38 <sup>(3)</sup>	Flat and Parallel	<u>1</u>
Total			20

(1) Containing a nominal 1.3 at % Nd.

(2) All rods were given dielectric end coatings for maximum reflectivity (>99.5%) at 1.06 $\mu$ .

(3) i.e., 1/4" x 1-1/2".

With the exception of one 3 mm x 50 mm rod shipped during the second quarter, all the remaining rods were fabricated from crystals grown toward the end of the program and which therefore reflect the advances in growth technique that were made. The next two sections summarize the results of the evaluation tests.

### 1. Passive Tests

All rods were first fabricated with flat and parallel ends so that the following passive optical evaluation procedures could be carried out on them:

- (1) Twyman-Green interferometer examination
- (2) Schlieren photographs
- (3) Beam divergence measurements
- (4) Far field photographs.

Figure 5 presents a schematic diagram of the optical bench layout utilized for obtaining the beam divergence values and far field photographs.

Some of these rods were then sent to an outside firm for application of hard dielectric coatings; the remainder had the ends refinished to 12" radius prior to coating. The coated rods were further evaluated in lasing tests described in the next section. Figures 6 through 18 summarize the results of the passive optical tests.\* The first two figures of this set describe 1/4 in. x 1-1/2 in. rods that were fabricated from the largest size-range crystals grown under this program similar to the one shown in Figures 1b, 2 and 3. As is evident from the Twyman-Green photographs and the schlieren photographs, both rods contained a small amount of the core (crystal diameters had still been a trifle small). However, because the cored regions

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\* In all cases the curves shown compare the measured passive beam divergence with the theoretical values for the diffraction limited case. The photographs show the results of the following tests, from left to right: Twyman-Green interferogram, far field photographs, schlieren photographs.

were closely confined to the outer peripheral areas of the rods in both cases, the effects on a plane wave front traversing the length of the rod were minimal. This localization of the optical distortion due to the eccentric position of the core inclusion is apparent not only in the Twyman-Green and schlieren photos but also in the results of the beam divergence tests and the far field photographs. Figure 8 shows similar evaluation data for a slightly smaller diameter but longer rod (5 mm x 50 mm) that was entirely free of core material. The schlieren photo shows an obvious improvement in the quality but the remaining passive test results are quite comparable to those obtained with the previous two rods.

The next three figures — 9, 10, and 11 — refer to rods of still smaller diameter, 3 mm x 50 mm, all of which were core-free. Nevertheless, the passive optical test results shown indicate the presence of a significantly greater amount of optical inhomogeneity than in the larger diameter rods. These results are interpreted to mean that sources of optical distortion other than the core are present, probably originating from less than optimal growth conditions prevailing in the smaller growth stations from which these latter crystals were obtained. Similar comments are equally applicable to the 3 mm x 75 mm rods for which the passive optical test results are shown in Figures 12 through 18. The quality of these rods per unit length is probably comparable on the average to that of the 3 mm x 50 mm rods but the cumulative effect of the additional length is clearly evidenced by the generally larger fringe count obtained in the Twyman-Green tests. Thus, the better of the 3 mm diameter rods show on the average 1 to 1-1/2 fringes per inch of length, in the Twyman-Green test. However, several of the 3 mm x 75 mm rods showed significantly higher fringe counts (e.g., Figures 14 and 16). No consistent correlation could be made between these high fringe counts and the appearance of the schlieren photographs; the rods appeared to be free of any obvious core inclusions. However, examination under crossed polars sometimes revealed a striated stress pattern that was usually more pronounced in the case of the high fringe-count rods. These stress patterns are thought to be associated with some sort of inclusions or scattering sites which in turn originate from sudden changes

in growth conditions. The large diameter rods, fabricated from crystals grown under more stable conditions, are relatively free of such disturbances.

## 2. Active Tests

The apparatus depicted in the schematic diagram of Figure 19 was utilized to determine pulsed thresholds. In this simple setup, the rods were mounted inside a helical xenon flashlamp contained in a MgO coated cylindrical chamber. A power supply was used to charge a capacitor bank. The instantaneous discharge of the capacitors fired the lamp. The output of the rod was detected by a E. G. G. SD-100 silicon photocell mounted at the end of the chamber and read out on an oscilloscope. CW tests were carried out in the specially designed elliptical head shown in Figure 20. The head was fabricated from aluminum. The interior was then coated with "Kanigan" (a proprietary coating), polished and lapped, and recoated with successive layers of vacuum evaporated metallic aluminum and silicon monoxide. The lamps used in this head were General Electric's Quartzline coiled tungsten filament model Q500T3/CL-120V. The power supply was designed to deliver from 0 to 800 watts (AC or DC). Laser output was determined in the same manner described for the pulsed head. Test results are summarized in Table II. Examination of these data clearly shows the influence of end configuration on threshold. Thus, in the case of the 3 mm x 50 mm rods, about a 41% increase in pulsed threshold occurred when the end configuration was changed from 12" radius to flat and parallel. Because the rods were fabricated from the same crystal (1540) and gave very similar results in the passive evaluation tests (see Figures 9 through 12), the increase is considered real and due entirely to the change in end configuration. Similar considerations apply to the 3 mm x 75 mm rods where an even larger increase occurred: 205%. However, in the case of the 6.35 mm x 38 mm rods, the threshold changed oppositely to what was expected: the rod with flat and parallel ends gave a threshold value about 10% less than that of a similar size rod with 12" radiused ends. The only explanation that presently seems reasonable is that the threshold of the 12" radiused rod (91A) was abnormally

high because of some defect not readily apparent from the results of the passive tests. Actually the passive test data were not found to be a consistently reliable guide to threshold performance. Thus, rod 1559B looked very poor in the passive tests (see Figure 14). It had the highest fringe count of any of the 3 mm x 75 mm rods and the far field pattern was so distorted that a meaningful set of beam divergence measurements could not be obtained. Nevertheless both the pulsed and CW threshold values obtained for this rod (see Table II) were nearly identical to values obtained for other rods adjudged to be of higher quality by virtue of their passive test results (e.g., rods 1561A, 1561B; see Figures 15 and 16).

TABLE II

LASER THRESHOLDS FOR Nd:YAG RODS DELIVERED TO MSFC \*

Rod No.	Size (mm)		Type of End Finish	Pulsed Threshold (joules)	CW Threshold (watts)	
	Diam.	Length			AC	DC
91A	6.35	38	12" Radius	4.00	---	---
103B	6.35	38	Flat and parallel	3.62	---	---
104C	5	50	Flat and parallel	4.41	---	---
1540A	3	50	Flat and parallel	3.62	---	---
1540C	3	50	12" Radius	2.57	540	560
1540D	3	50	12" Radius	2.57	580	600
1557A	3	75	12" Radius	2.57	570	585
1557B	3	75	Flat and parallel	7.29	---	---
1559B	3	75	12" Radius	2.89	615	630
1561A	3	75	12" Radius	2.89	620	640
1561B	3	75	12" Radius	2.89	645	655
1564A	3	75	12" Radius	2.57	575	590
1564B	3	75	Flat and parallel	7.84	---	---

\* All rods coated on both ends with dielectric coatings to >99.5% reflectivity at  $1.06\mu$ .

The data in all cases are for room temperature operation.



#### IV. CONCLUSIONS

##### A. Growth

The stations used for the growth of Nd:YAG crystals have been scaled up, redesigned, and improved by stages during the course of this program. These advances in technique have made possible the growth of ever larger Nd:YAG crystals. Thus, although at the start of this program, the largest core-free Nd:YAG rods which could be routinely produced with reasonably consistent high quality were 3-mm diameter x 50-mm long, at the present time crystals can be grown in a variety of larger size ranges to yield rods of the following dimensions: 3 mm x 75 mm, 5 mm x 50 mm, 6.35 mm x 38 mm. However, the growth of the largest size crystals, 0.65-0.75 inch in diameter by 3 to 3-1/2 inches long that are required for the fabrication of 1/4-inch diameter core-free rods still requires additional development work in order to optimize the process. The most pressing concern at the moment is system reliability. For reasons fundamentally associated with the unfavorable size relationship of the  $\text{Nd}^{+3}$  ion to the available site size in the YAG lattice, Nd:YAG crystals must be grown at very slow rates in order to achieve high quality. Therefore, as crystal size requirements are increased, so do total run times stretch out proportionately. This means that the probability of failure or malfunction of some critical component in the growth station is also increasing. Moreover, since a priori identification of these weak links was not possible, the only practical alternative course of action was to carry out relatively large numbers of long runs so as to force all unreliable components to fail or unequivocally demonstrate some progressively deleterious effects on crystal quality. System operation could then be decisively improved by replacing the defective component or by suitable alterations in system design. In this fashion the overall performance of the growth stations has been progressively upgraded.

##### B. Effects of Annealing

The work carried out under this program has conclusively demonstrated that the optical inhomogeneities associated with the cored region in as-grown crystals cannot be eliminated by annealing. Furthermore, annealing was not found to produce

any significant improvements in core-free fabricated rods, as judged by passive optical tests. These latter results are thought to be related to the presence of certain types of microscopic inclusions, either bubbles or solid particles, which cause localized stresses in the lattice and associated optical distortion. Unless the annealing causes elimination of these discontinuities, no significant improvements in optical quality can be expected via this approach. This means that additional gains in quality can apparently best be achieved by further advances in growth technology.

### C. Evaluation

Both passive optical and active lasing tests indicate that the quality of the larger crystals compares very favorably with that of the smaller sizes. Indeed there is a strong indication that the growth conditions utilized to produce the largest diameter Nd:YAG crystals favor the formation of very high quality material for two reasons primarily: (1) these crystals are grown in the scaled-up stations from larger melt volumes which tend to enhance thermal stability, (2) the increase in crystal diameter can be accomplished with only a minor increase in the cross-sectional area occupied by the core. This means that the disturbing influence of the core on the optical homogeneity of the surrounding material is correspondingly diminished.

The active tests showed a clear relationship between threshold and type of end configuration applied to the rods: thus the rods with radiused ends normally displayed significantly lower threshold values than similar size rods with flat and parallel ends. By the same token, however, it would be anticipated that active beam divergence would increase with increasing radius of curvature on the ends. Thus, the user must decide what constitutes an acceptable trade-off between these two effects for any specific application.

Comparison of passive test results with threshold values for each rod does not reveal any striking correlations. Passive tests are not, therefore, a good guide to threshold performance.

## V. RECOMMENDATIONS

The present program has served the valuable and necessary function of promoting the development of Nd:YAG to a point where its laser potentialities may be more fully and realistically assessed in a given area of application. However, although real progress has been made in the past year in improving both the growth process and the crystal quality, it is also recognized that the present level of technological know-how must be advanced considerably further in order to permit the most efficient production and effective utilization of Nd:YAG. At least two major areas may be distinguished where additional R and D work appears to be most needed in the near future:

1. Optimization of techniques used for the growth of larger size Nd:YAG crystals.

The available evidence strongly indicates the gains in quality to be achieved by growing large size Nd:YAG crystals. The significance of this finding is that regardless of whether the ultimate user requires a 3 mm x 30 mm rod or a 7 mm x 100 mm rod, from the standpoint of quality, it will be most desirable and advantageous to fabricate all such rods from the largest size crystals that can be grown. However, considerable refinement of the presently utilized growth process will be required in order to permit routine production of these larger crystals with uniform quality and in good yields. Additional development work in this area is definitely indicated.

2. Better correlation of passive and active performance.

Although there are some obvious relationships between crystal quality and lasing behavior, a distinct need exists for more quantitative correlations between quality as measured by various passive tests and the lasing performance. Acquisition of this knowledge would be of great benefit not only to the ultimate user, but would provide invaluable guidance to the crystal grower as well. Expanded R and D work in the field of laser testing of Nd:YAG in a setting that was closely allied to growth activity would seem, therefore, to be a very desirable objective.

From a larger range point of view further attention should be given to the problem of finding a growth technique that would yield core-free Nd:YAG crystals. Although the presence of the core in the as-grown crystals no longer poses as great a problem as it once did in view of the successful development of methods for growing larger size crystals in which the core region can be confined to a relatively small fraction of the cross-section, nevertheless its elimination would be desirable since then the entire cross-section of the crystal could be utilized and, in addition, the danger of random strain flares would be considerably reduced. However, present knowledge suggests that rather a drastic departure from the currently utilized Czochralski technique might be required to achieve this result. Hence further exploratory development work in this area will be necessary.

## VI. PROGRESS RATE AND FUND EXPENDITURES

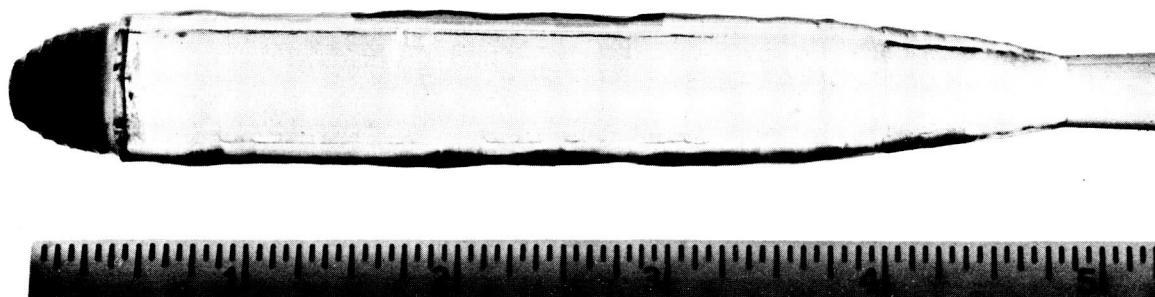
The progress rate and fund expenditures throughout the lifetime of this program are shown in Figure 21. The expenditure limit specified in the contract was reached in March 1966 although some additional work remained to be performed in order to fulfill the contract objectives. The stretchout in the work schedule was necessitated by the desire to provide MSFC with the highest possible quality Nd:YAG rods that would truly reflect the advances in growth techniques made during the course of the program. Therefore, selection of material for fabrication of the final 15 rods was delayed as long as possible in an attempt to reap the maximum benefit from the optimization studies that were in progress. Consequently some of the fabrication work, end coating, and evaluation and, of course, preparation of this final report, extended beyond the scheduled completion date. However, no further charges were levied against the contract beyond the month of March.

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(a)



(b)

Figure 1 Typical as-grown appearance of Nd:YAG crystals in various size ranges.

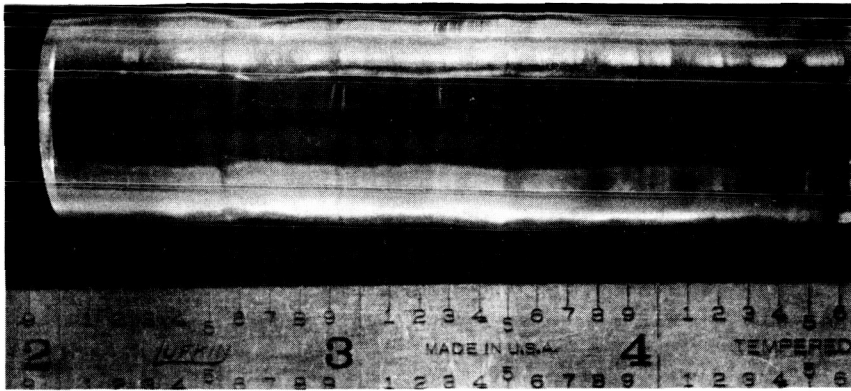


Figure 2      Appearance of core region under crossed polars of crystal shown in Fig. 1b.

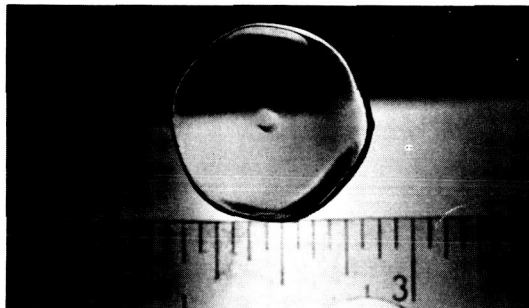


Figure 3      End view of core region under ordinary light.  
(Same crystal as in Fig. 1b.)



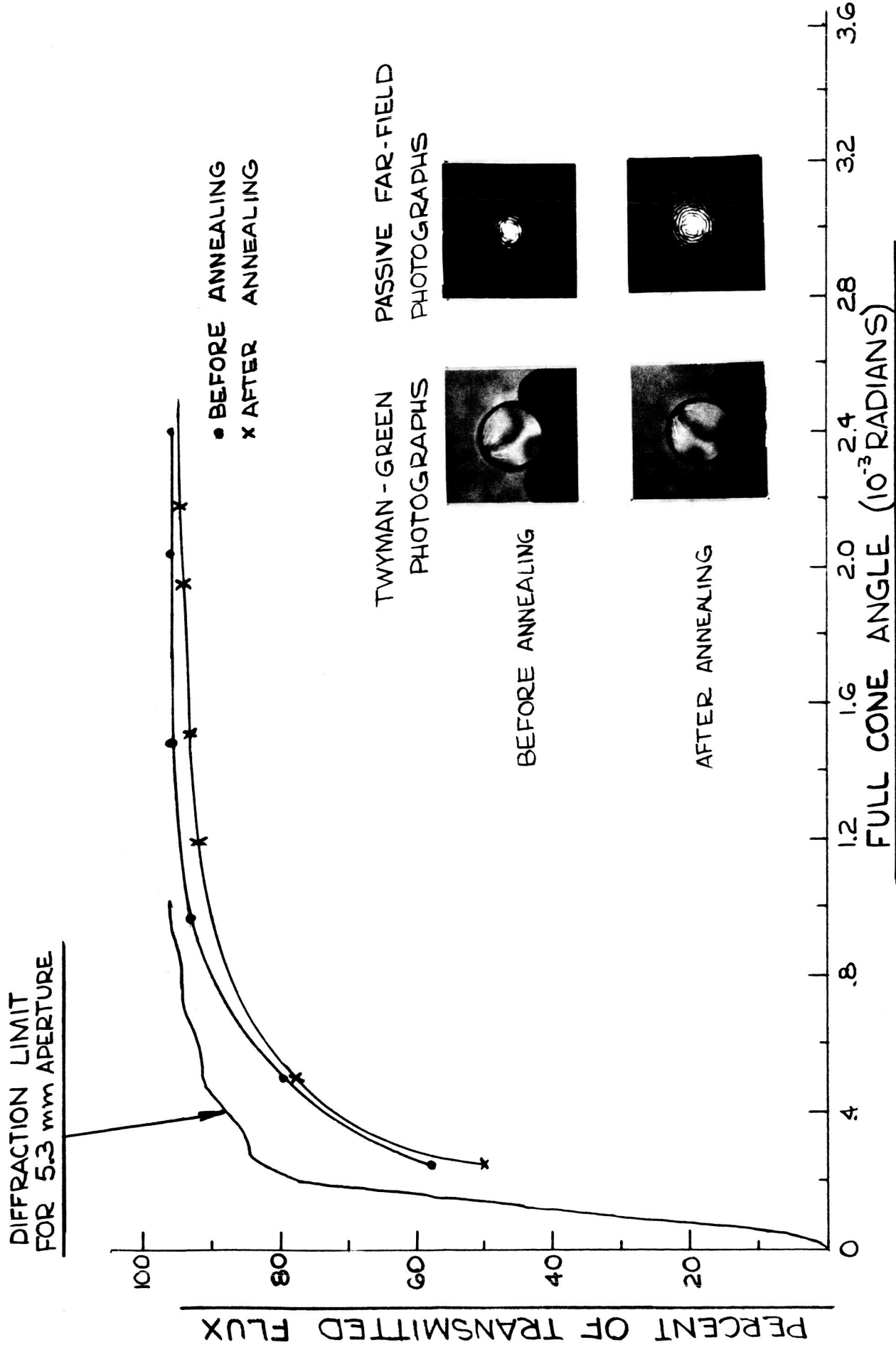
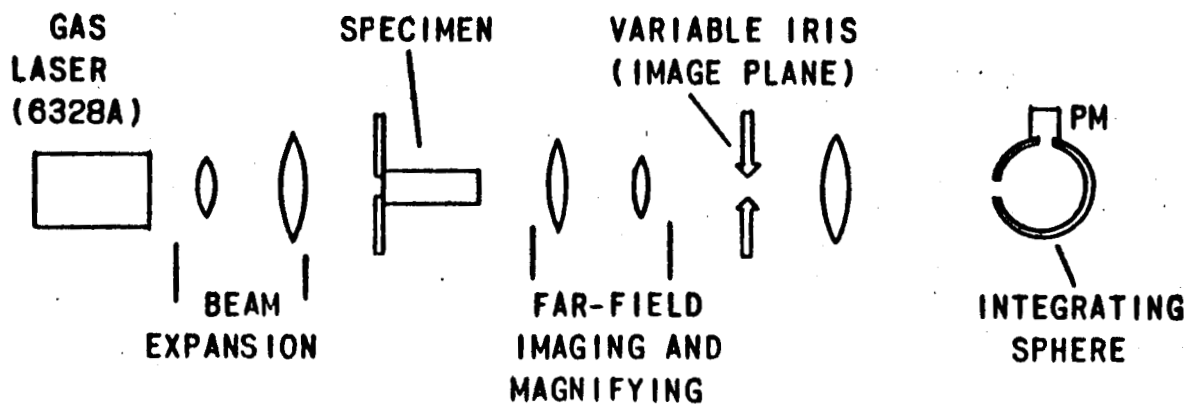


FIG.4 RESULTS OF 41 HR. ANNEAL AT 1850-1870°C. ROD NO. 1493A



**FIGURE 5 APPARATUS FOR MEASURING THE FAR-FIELD ENERGY FLUX DISTRIBUTION.**

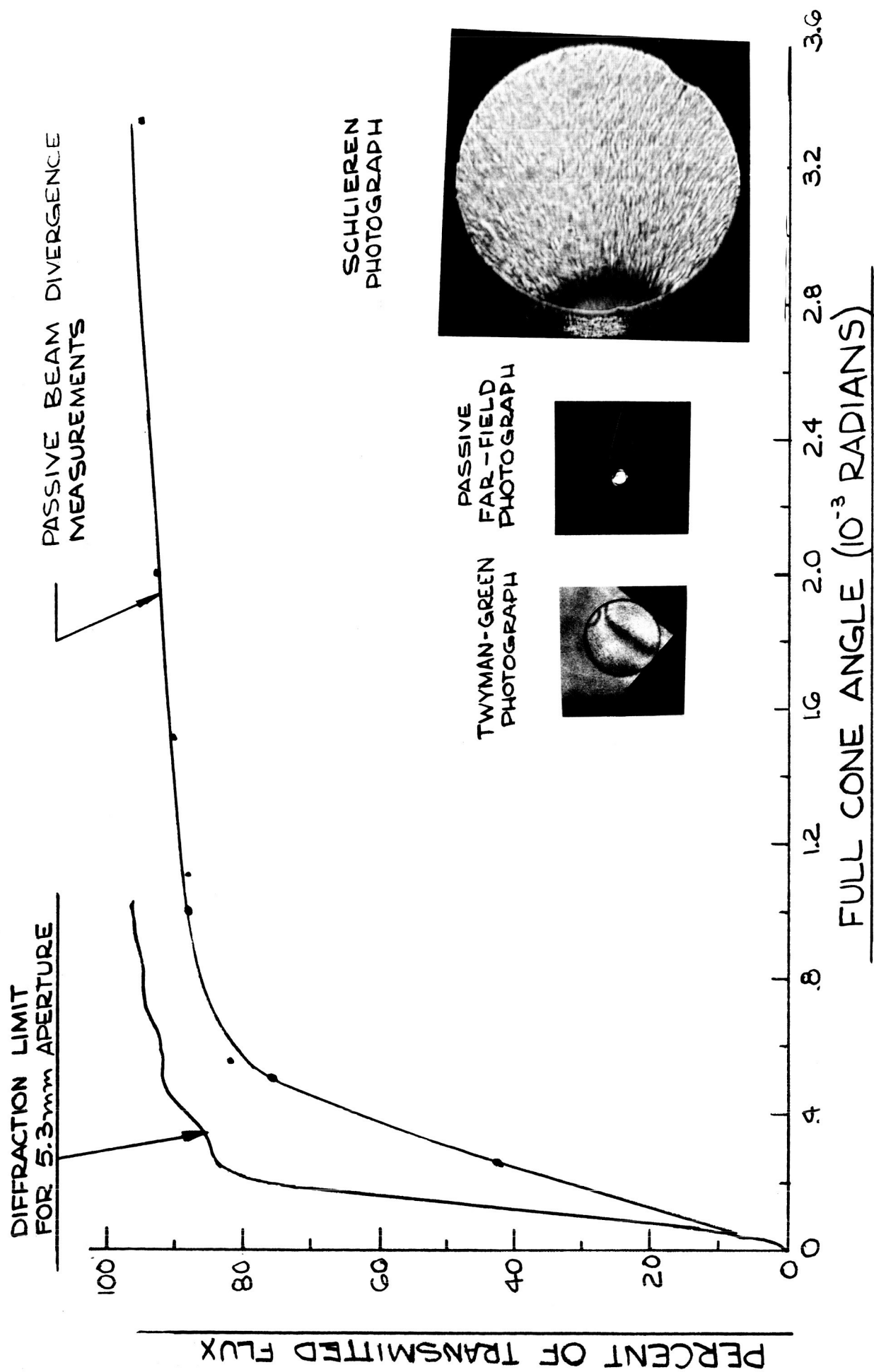


FIG. 6 ROD NO. 91A. 6.38 mm x 38 mm

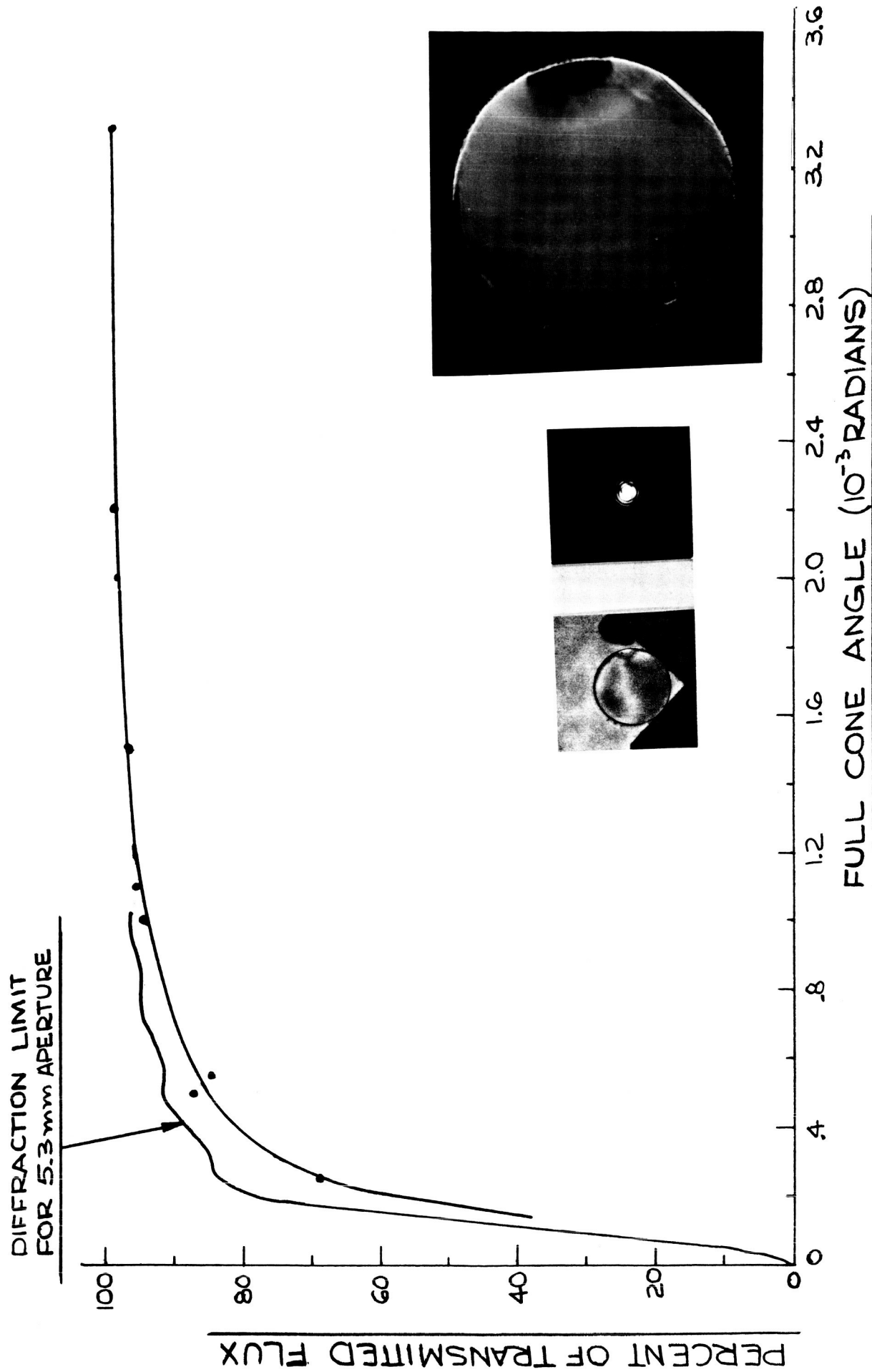
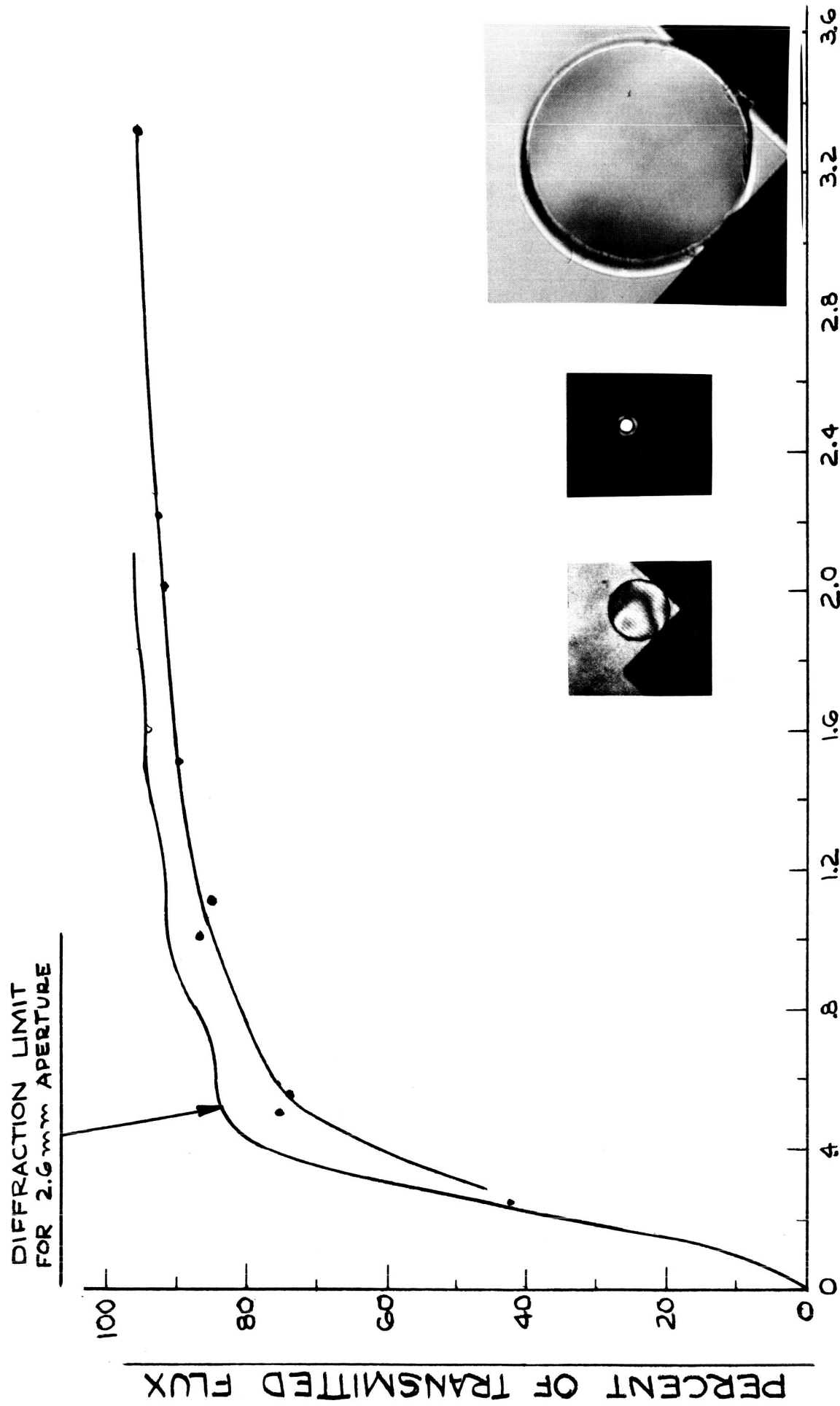


FIG. 7 ROD NO. 103 B. 6.38 mm x 38 mm



FULL CONE ANGLE ( $10^{-3}$  RAD.)

FIG. 8 ROD NO. 104C. 5 mm x 50 mm

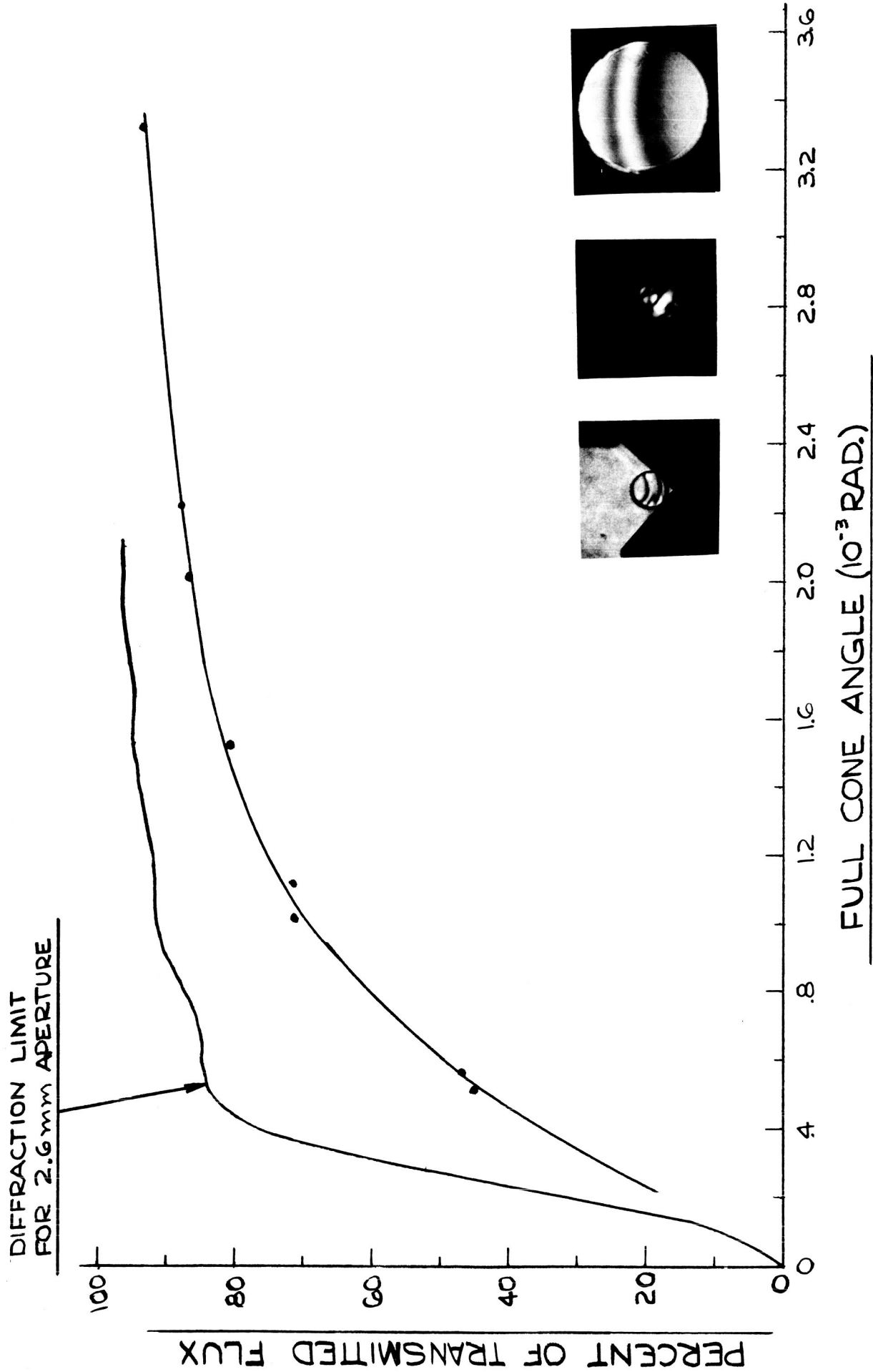
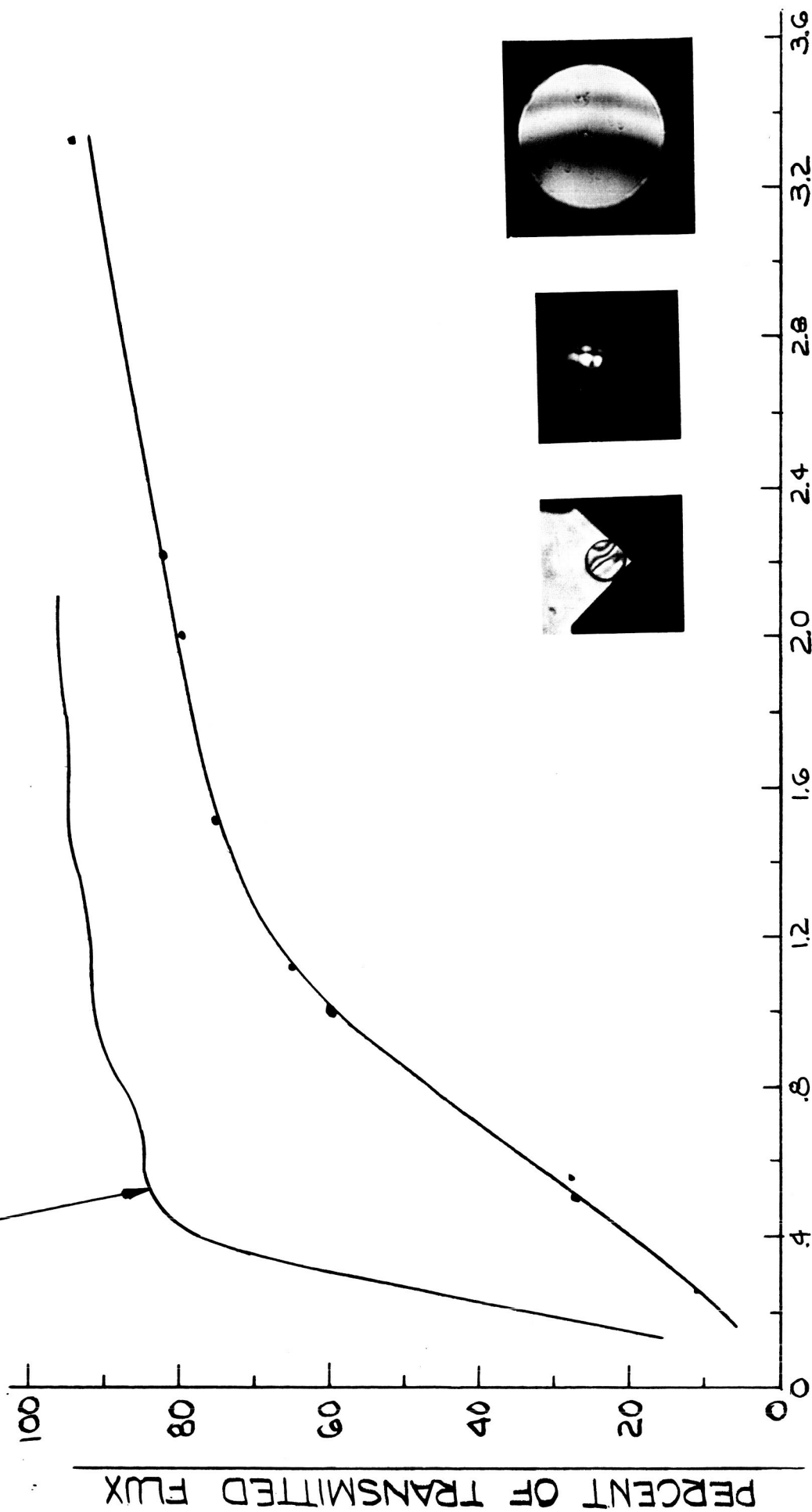


FIG. 9 ROD NO. 1540A. 3mm X 50mm.

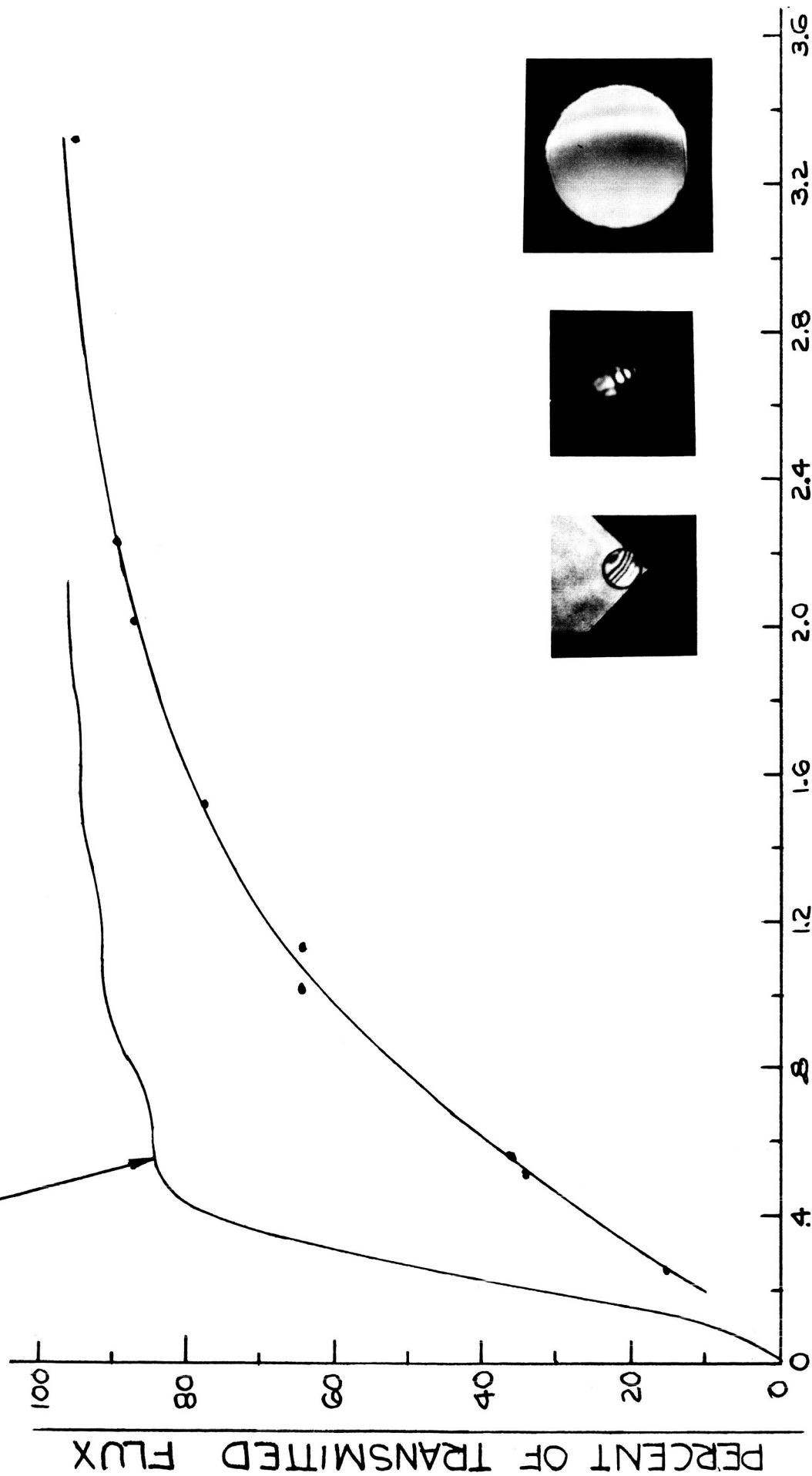
DIFFRACTION LIMIT  
FOR 2.6mm APERTURE



FULL CONE ANGLE ( $10^{-3}$  RAD.)

FIG. 10 ROD NO. 1540C. 3mm x 50mm

DIFFRACTION LIMIT  
FOR 2.6 mm APERTURE

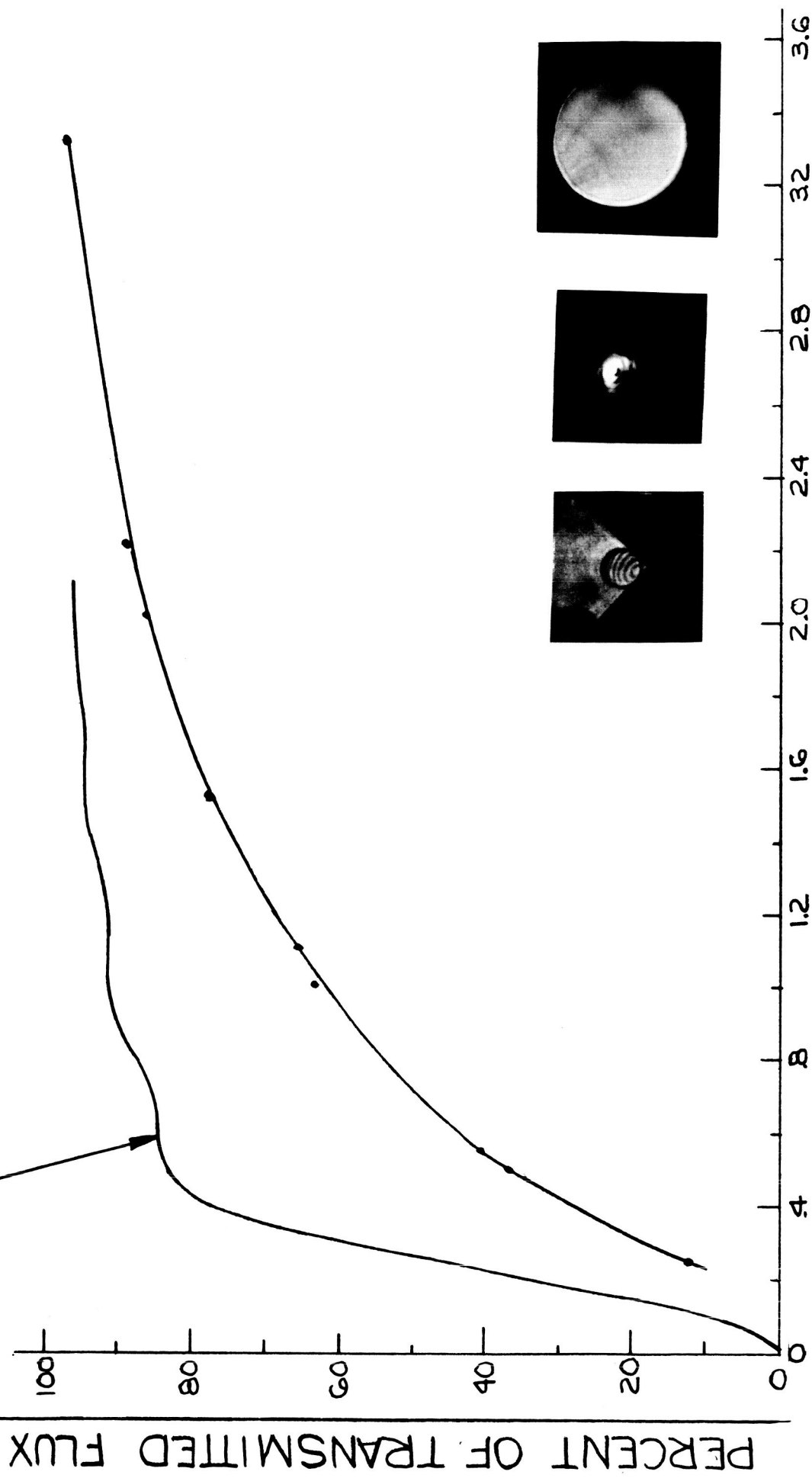


FULL CONE ANGLE ( $10^3$  RAD.)

FIG. 11 ROD NO. 1540D. 3mm x 50mm

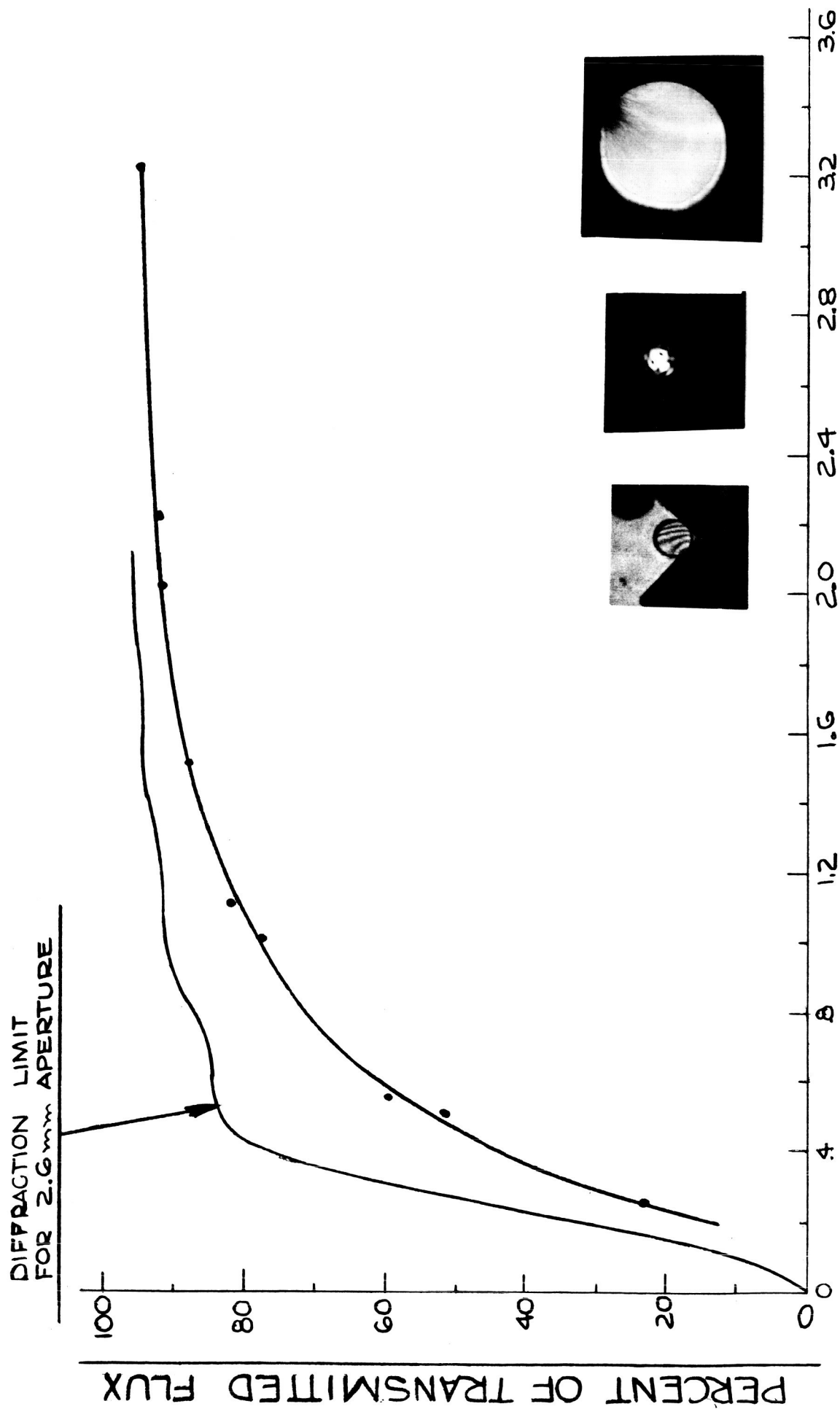


DIFFRACTION LIMIT  
FOR 2.6 mm APERTURE



FULL CONE ANGLE ( $10^{-3}$  RAD.)

FIG.12 ROD NO. 1557 A. 3 mm x 75 mm

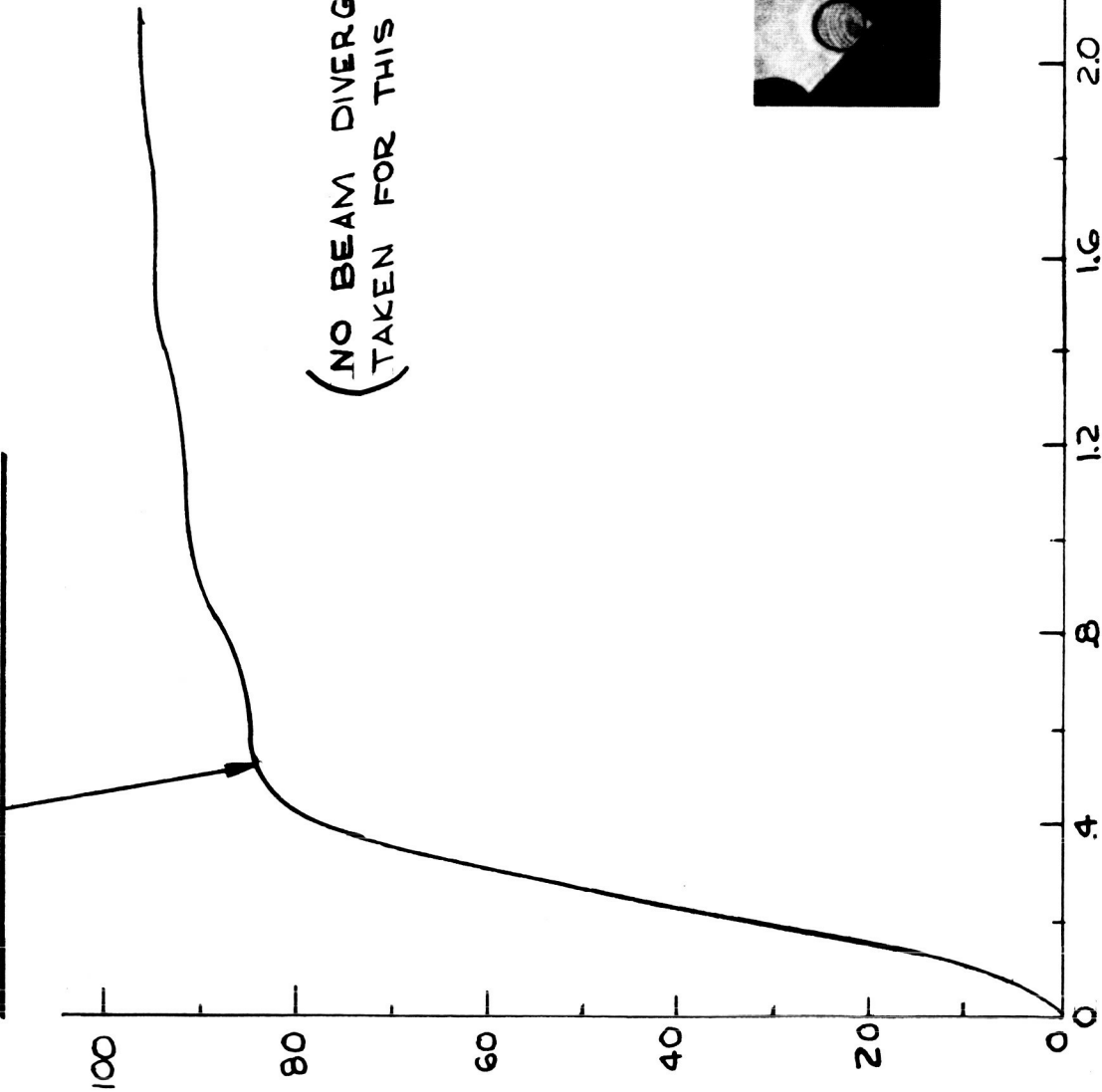


FULL CONE ANGLE ( $10^{-3}$  RAD)

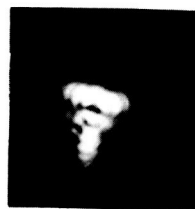
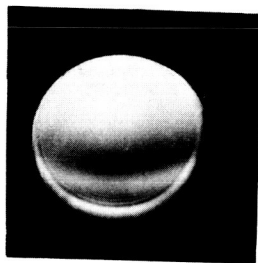
FIG. 13 ROD NO. 1557 B. 3mm X 75mm

DIFFRACTION LIMIT  
FOR 2.6 mm APERTURE

PERCENT OF TRANSMITTED FLUX



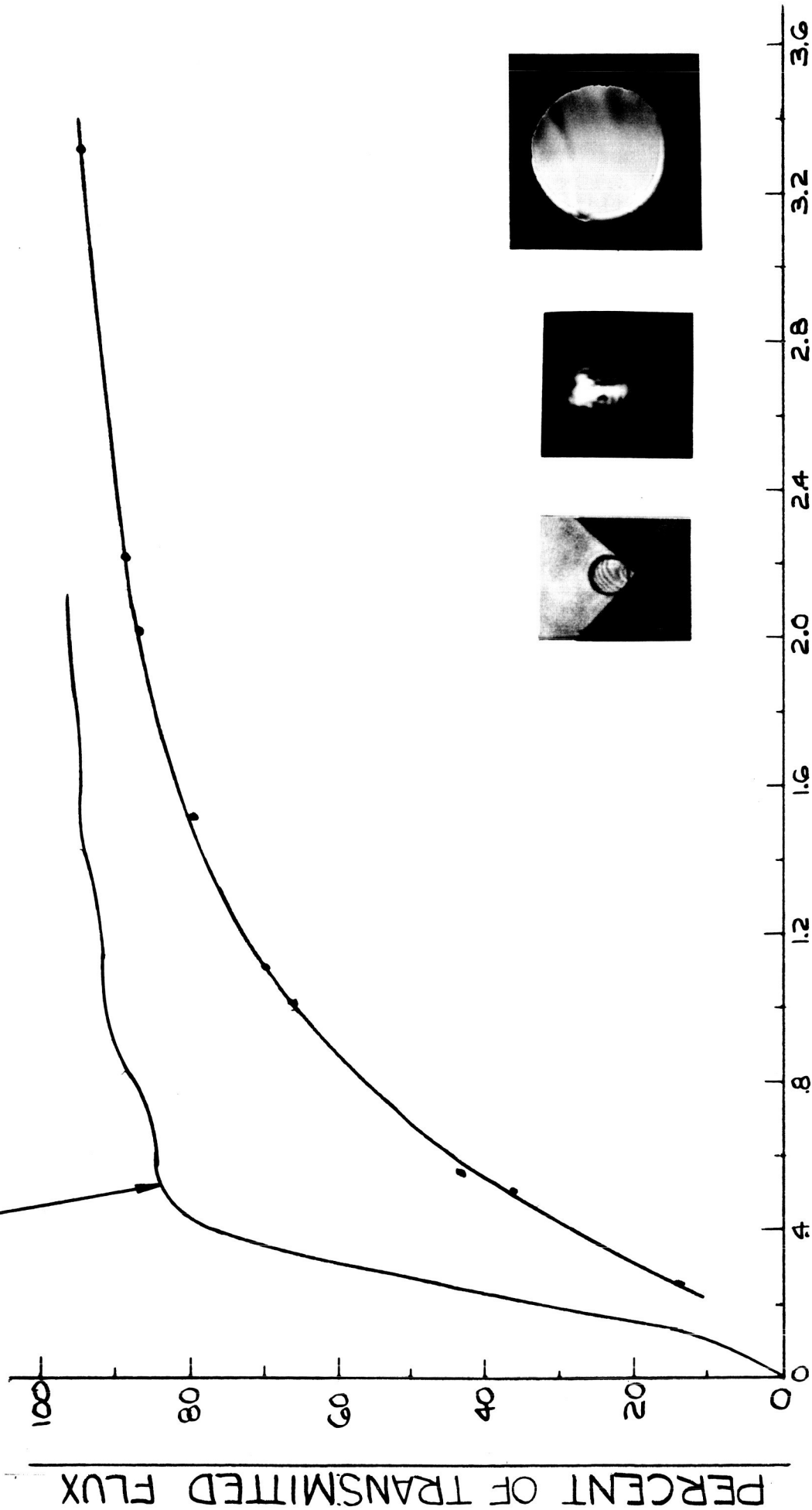
(NO BEAM DIVERGENCE MEASUREMENTS)  
(TAKEN FOR THIS ROD)



FULL CONE ANGLE (10<sup>-3</sup> RAD.)

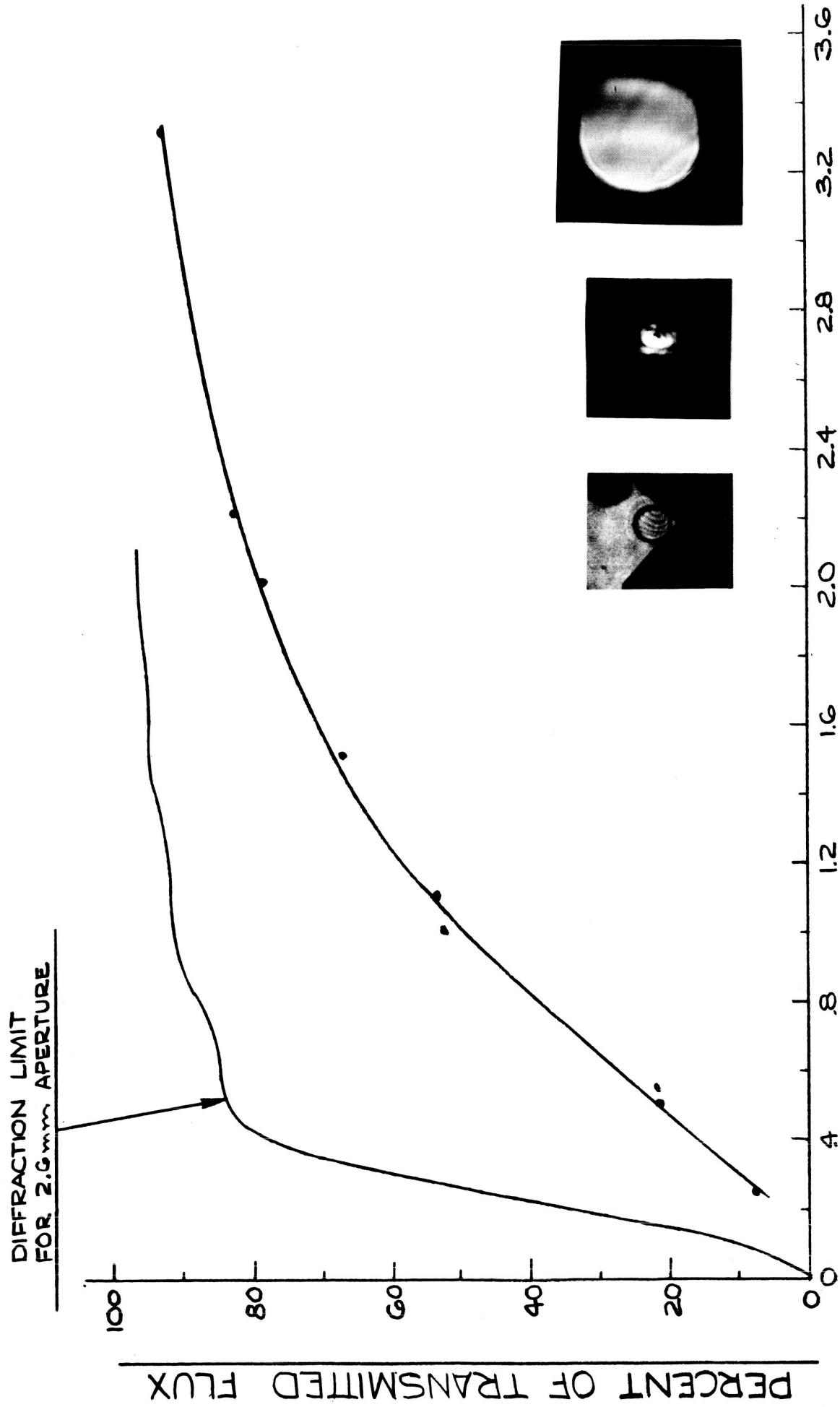
FIG. 14 ROD NO. 1559 B. 3 mm x 75 mm

DIFFRACTION LIMIT  
FOR 2.5mm APERTURE



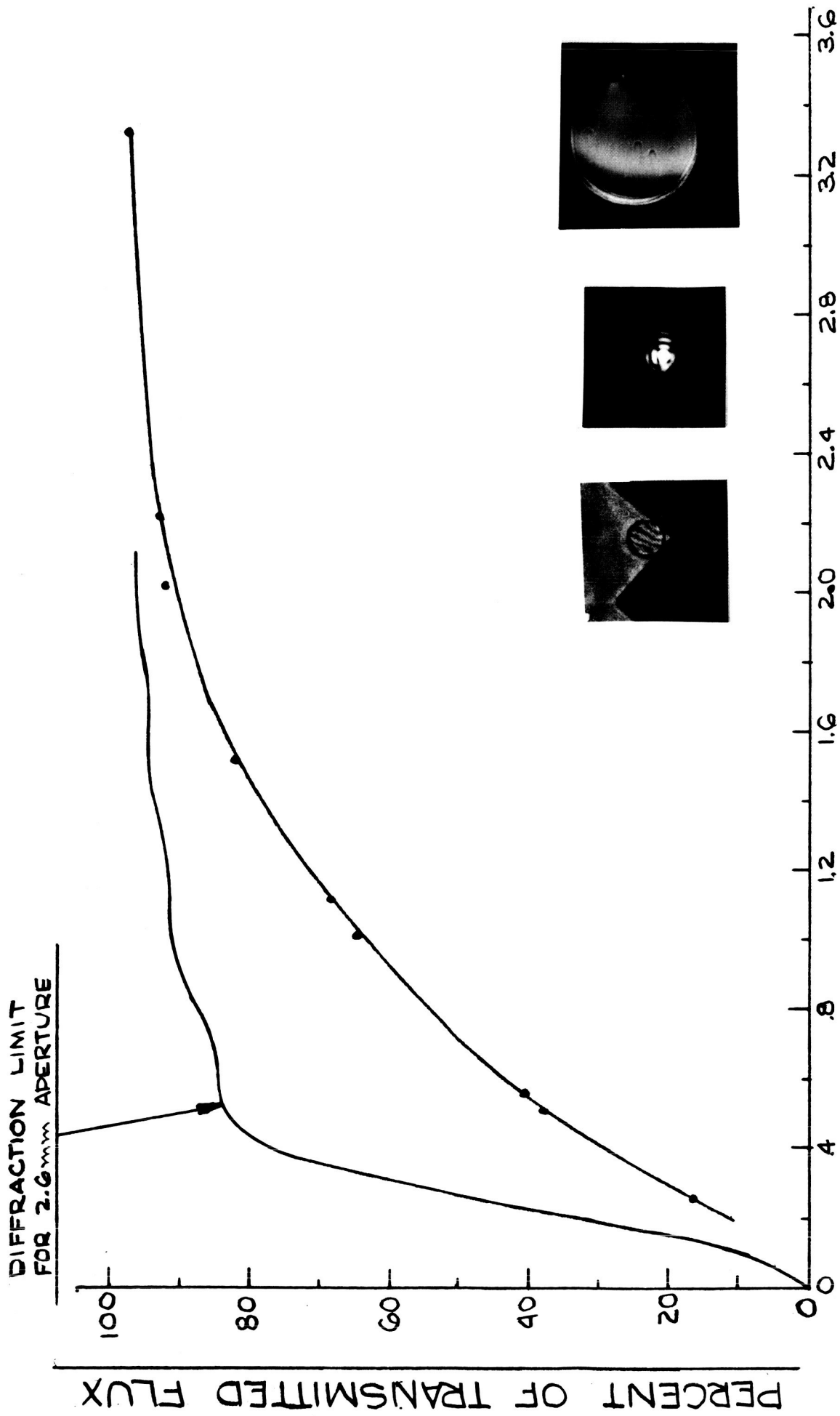
FULL CONE ANGLE ( $10^{-3}$  RAD.)

FIG. 15 ROD NO. 1561A. 3mm X 75mm



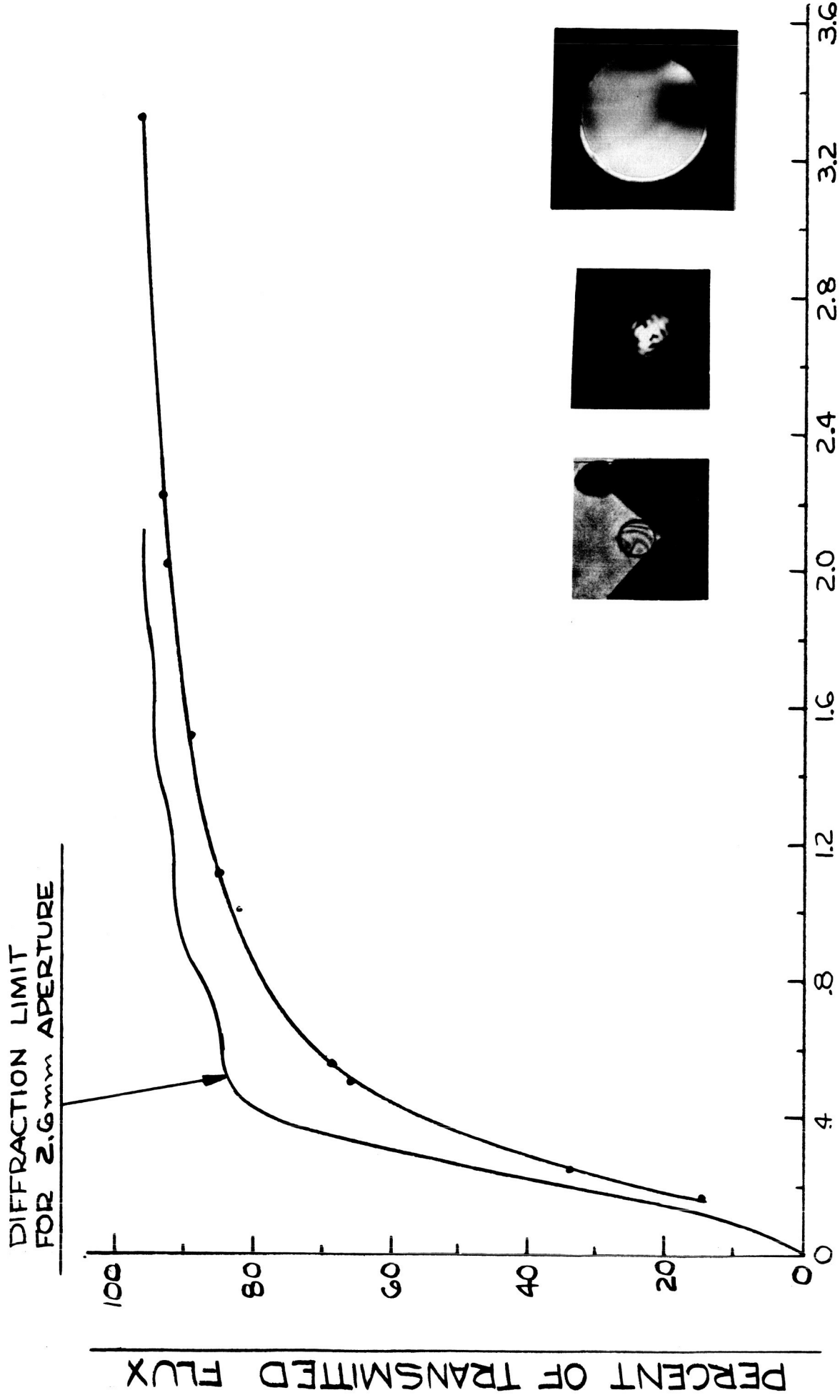
FULL CONE ANGLE ( $10^{-3}$  RAD.)

FIG.16 ROD NO.1561 B. 3mm X 75mm



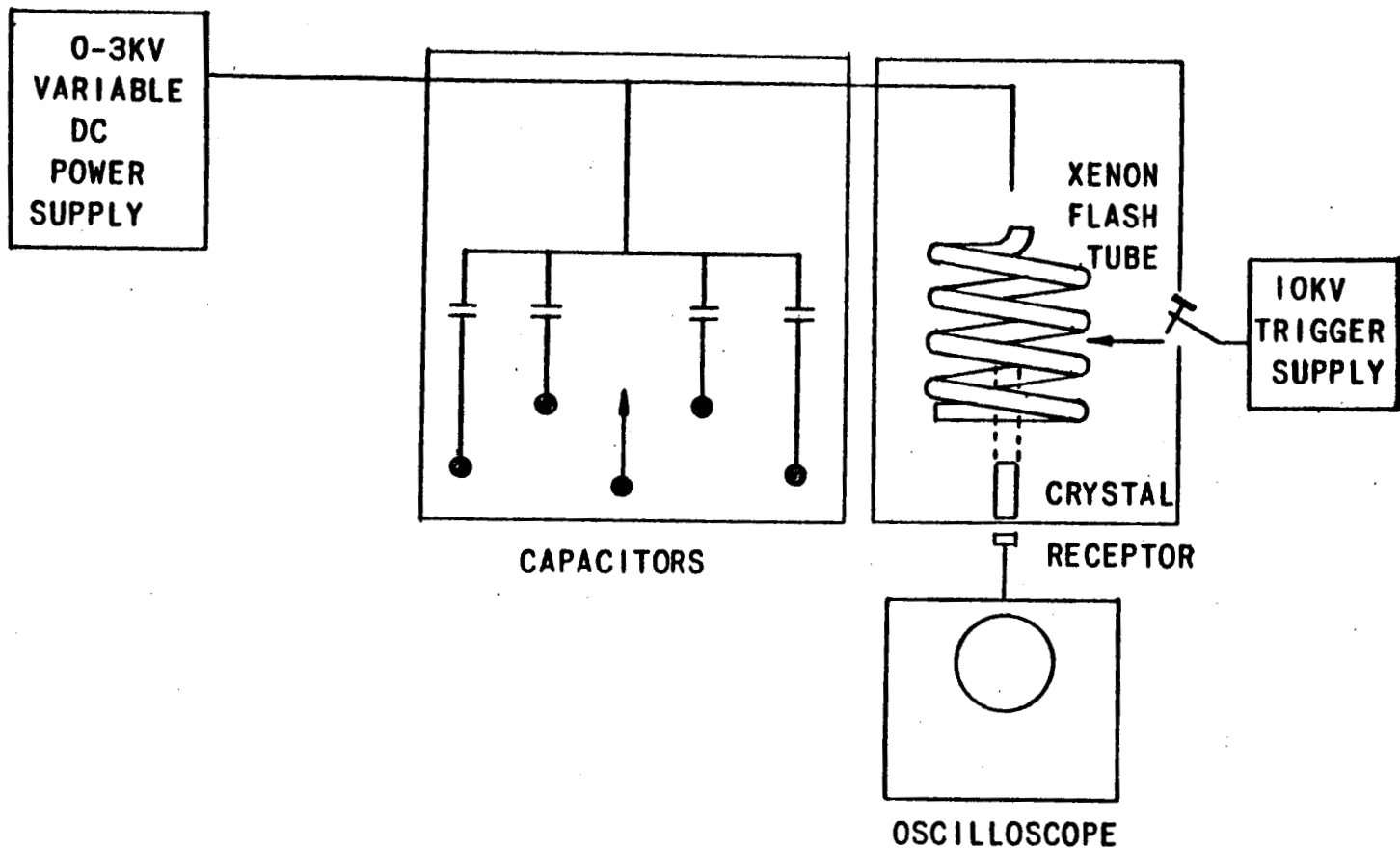
FULL CONE ANGLE ( $10^{-3}$  RAD.)

FIG. 17 ROD. NO. 1564A. 3mm X 75mm



FULL CONE ANGLE ( $10^{-3}$  RAD.)

FIG. 18 ROD NO. 1564 B. 3mm X 75mm



CAPACITORS-.5, 2, 4, & 8 $\mu$ fd.

FLASH TUBE-GE FT-524 IN MgO REFLECTOR

RECEPTOR-E.G.G. SD-100 SILICON PHOTO-CELL

FIGURE 19-LINDE COHERANCE DETECTOR.



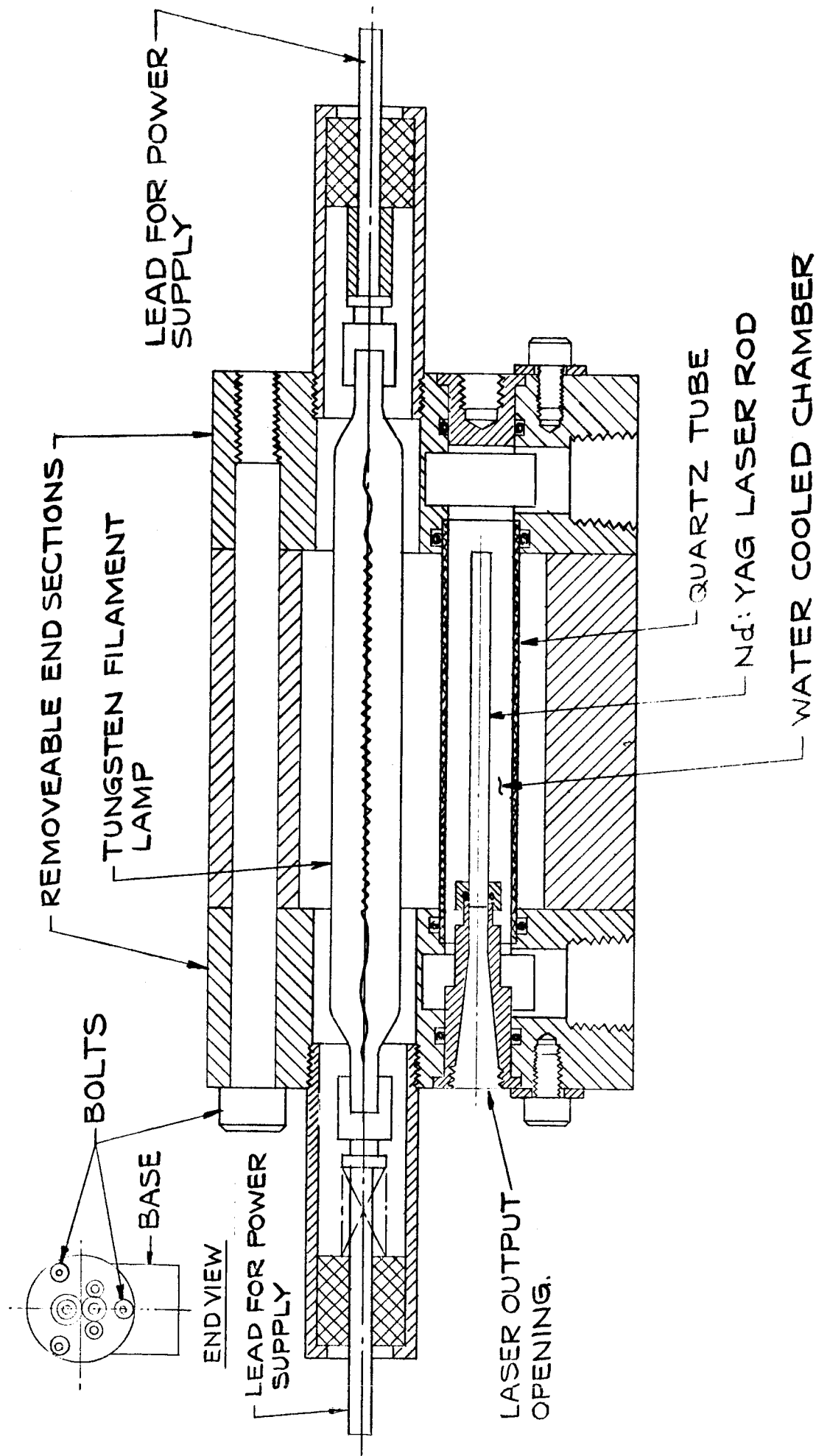


FIGURE 20 SCHEMATIC DIAGRAM OF CW TEST HEAD

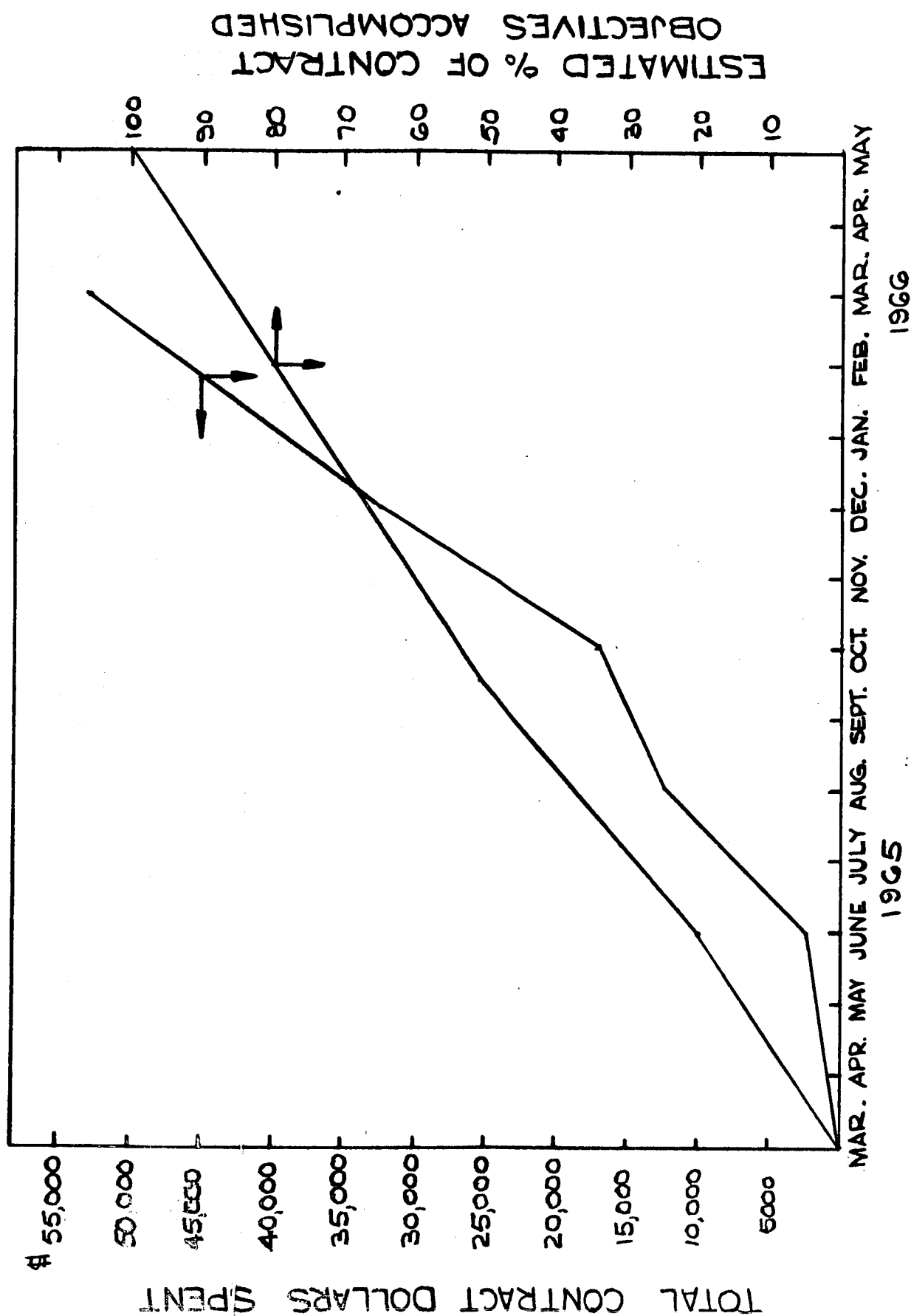


FIG. 21 PROGRESS RATE AND FUND EXPENDITURES